SYNOPSIS

Interaction Between Polyelectrolytes and Surface Active Ionic Liquids

Thesis submitted for the degree of
Doctor of Philosophy in Science (Chemistry)
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Synopsis of the Thesis

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Polyelectrolytes constitute a special class of polymers with dissociable groups which ionize in polar media (*e.g.*, water) to yield polyions and counterions bearing opposite charges. Polyelectrolytes have been and continue to be a very active area of scientific research and commercial growth. This ubiquitous class of materials finds widespread application in chemistry, physics, biology, medicine, materials science, and nanotechnology.[1-17] In many domestic, industrial and technological applications polyelectrolytes are coupled with conventional surface active agents (surfactants). The interaction between polyelectrolytes and surfactants plays an important role in such areas as detergency, photographic materials, coatings, cosmetics, drug delivery, enhanced oil recovery, paints, pharmaceuticals, biomedical application, food and mineral processing.[13-15] Consequently, the interactions between them have been extensively investigated over the past decades.[13-15]

Due to the increasing consciousness of environmental problems, chemical processes are now being developed keeping their environmental burden in mind. This results in an upsurge of green chemistry encouraging the design of products and processes that reduce or eliminate the use and generation of hazardous substances. In this context, ionic liquids figure prominently because of their special properties, such as low volatility, tunable viscosity and miscibility, non-flammability, non-corrosivity, high thermal stability, high ionic conductivity, high solvating capacity for organic, inorganic, organometallic and polymeric materials.[18-24] Recently, some ionic liquids comprising of a pronounced hydrophilic and hydrophobic molecular segments have been shown to exhibit surface activity like the conventional ionic surfactants. This type of ionic liquids is commonly referred to as surface active ionic liquids. The self-assembly of surface active ionic liquids in pure states and in solutions is confirmed by experiment and computer simulations.[25] Considerable progress has been made on the studies of micellization of surface

active ionic liquids in aqueous solutions.[26-40] The self-assembly of surface active ionic liquids may have serious impact in extraction and chromatography.[41-43] The tendency of surface active ionic liquids to self-organize in solutions opens up broad prospects of their application. Surface active ionic liquids can effectively replace the conventional surfactants in many applications *e.g.*, in manufacturing nano-structured materials, in solubilization, protein folding, drug delivery etc.[44-49] Thus, surface active ionic liquid properties opens up, in principle, the possibility to use mixtures of polyelectrolytes and surface active ionic liquid in areas where polyelectrolyte-conventional surfactant combinations are used now-a-days. Polyelectrolytes can modulate the behaviour of surface active ionic liquids through diverse interactions depending upon polyelectrolyte sample, its concentration, temperature etc. It is also possible to "engineer" the polyelectrolyte-surface active ionic liquid interactions by a judicious choice of the ionic constituents of the surface active ionic liquids. The fact that the polyelectrolyte-surface active ionic liquid interactions can be controlled by a varying number of parameters makes them not only an interesting and rich area for the exploration of novel phenomena but also an area of research which can lead to new applications in a variety of fields.

With a view to elucidate the polyelectrolyte-surface active ionic liquid interactions, the present dissertation aims to investigate the influence of polyelectrolytes on the aggregation behaviour of surface active ionic liquids in aqueous solutions using different experimental techniques (conductometry, tensiometry, osmometry, and spectrofluorimetry). The counterion-condensation behaviour of polyelectrolytes in absence of surface active ionic liquids, and ion-association and aggregation behaviour of surface active ionic liquids in absence of polyelectrolytes have also been explored to help unravel the nature of interactions in polyelectrolyte-surface active ionic liquid solutions. The overall objective is to provide a comprehensive understanding of polyelectrolyte-surface active ionic liquid interactions keeping in view that studies exploring these interactions are still in their infancy.[50-54]

The main factors operative in solutions where both polyelectrolyte and surface active ionic liquid are present together are electrostatic interactions between the charged groups on polyelectrolyte chain and the surface active ionic liquid head groups, and the hydrophobic interactions distributed along the apolar tails of the surface active ionic liquid molecules. Such interactions

lead to aggregation and complex formation. The resulting complex differs in its conformational, structural and dynamical features from that of a polyelectrolyte or a surface active ionic liquid. A thorough understanding of the effects of the type and concentration of polyelectrolyte, alkyl chain length of the surface active ionic liquid, temperature etc. on the aggregation behaviour of surface active ionic liquids should provide important insight into the polyelectrolyte-surface active ionic liquid interactions.

The scope of this work covers the assessment of the counterion-condensation behaviour of polyelectrolytes, the aggregation behaviour of the surface active ionic liquids, and of the processes involved in the formation of various species (*surface-active* complexes consisting of surfactant monomers bound to the polyion backbone, *non-surface active* necklace-like polyion-micellar aggregates, and free surfactant micelles) in solutions containing both polyelectrolyte and surface active ionic liquid.

The interactions between the polyelectrolytes and the surface active ionic liquids were not explored till January 2012 when this study was undertaken. Besides two publications on NaCMC-C_{10}MeImCl and NaCMC-C_{16}MeImCl derived from this study,[55,56] a few papers have, so far, been published on different PE-SAIL systems by other groups.[50-54]

In order to understand the nature of polyelectrolyte - surface active ionic liquid interactions in aqueous solutions, in particular, and that of polyelectrolyte-surfactant systems, in general, a comprehensive multi-method approach would be suitable. With this objective, we performed a study using conductometric, tensiometric, osmometric and spectrofluorimetric techniques to probe various interactions prevailing in these solutions.

The aim of this thesis is to provide a better understanding about interactions between polyelectrolytes and oppositely charged surface active ionic liquids: What are the parameters affecting these interactions and how the subtle interplay of various forces determine their physico-chemical properties. Another important part of this study was to clarify the nature of interactions in pure polyelectrolyte and pure surface active ionic liquid solutions and how these interactions might be modified in each others presence.

For the study, we selected two negatively charged polyelectrolytes – sodium carboxymethylcelulose and sodium polystyrenesulfonate, and two surface active ionic liquids – 1-decyl-3-methylimidazolium chloride and 1-hexadecyl-3-methylimidazolium chloride. Four different experimental techniques, namely, conductometric, tensiometric, osmometric and spectrofluorimetric techniques were employed to explore and elucidate the nature of interactions in the investigated polyelectrolyte-surface active ionic liquid solutions.

Specifically, the wok embodied in this thesis includes:

1. Investigation on the counterion-condensation behaviour of the polyelectrolytes sodium carboxymethylcelulose and sodium polystyrenesulfonate in aqueous solutions.

2. Investigation on the ion-association behaviour of the surface active ionic liquids1-decyl-3-methylimidazolium chloride and 1-hexadecyl-3-methylimidazolium chloride in aqueous solutions in the premicellar regime.

3. Investigation on the aggregation behaviour of the surface active ionic liquids 1-decyl-3-methylimidazolium chloride and 1-hexadecyl-3-methylimidazolium chloride in aqueous solutions.

4. Investigation on the aggregation behaviour of the aqueous surface active ionic liquids (1-decyl-3-methylimidazolium chloride and 1-hexadecyl-3-methylimidazolium chloride) induced by the polyelectrolytes (sodium carboxymethylcelulose and sodium polystyrenesulfonate).

Both sodium carboxymethylcelulose and sodium polystyrenesulfonate are important industrial polymers with a wide spectrum of applications. Besides exploring the interactional features in the investigated polyelectrolyte-surface active ionic liquid systems, the present study is, therefore, expected to help extend the fruitful use of these polyelectrolytes.

Chapter 1 provides a general introduction to polyelectrolytes and their counterion-condensation behaviour in solutions, surface active ionic liquids and their interfacial and micellization properties, and polyelectrolyte-surface active ionic liquid interactions. A brief review of the

notable works in the fields of polyelectrolyte solution chemistry with particular reference to the condensation of counterions onto the polyions and those in the fields of surface active ionic liquids and polyelectrolyte-surface active ionic liquid mixed systems have been given. This is followed by an account of conductometry, tensiometry, osmometry, and spectrofluorimetry to investigate the interactions between polyelectrolytes and surfactants with special reference to the surface active ionic liquids has been given since these techniques have been employed to study the behaviour of polyelectrolyte-surface active ionic liquid interactions in this dissertation.

Chapter 2 describes the electrical conductivities of aqueous solutions of the anionic polyelectrolyte sodium carboxymethylcelulose with different degrees of substitution as a function of concentration and temperature. It gives a description of an investigation on the thermodynamic, transport and frictional properties of sodium carboxymethylcelulose in aqueous solution. The conductance data have been analyzed on the basis of a simple equation with only one adjustable parameter following the model for the electrical conductivity of solutions of semidilute polyelectrolytes without added salt proposed by Colby *et al*.[57] using a scaling description for the configuration of a polyion chain according to Dobrynin *et al*.[58] Excellent agreement with the experimental results were obtained. 36–55% of the counterions were found to remain condensed and the condensation process was shown to be spontaneous. Although the level of counterion-condensation depends appreciably on the degree of substitution of the polyelectrolyte, it was found to remain invariant with the polyelectrolyte concentration and the temperature. The free energies of counterion-condensation, the ratios of the mobility of the polyion to that of the counterion, the polyion transference numbers, and the coefficients of monomer-solvent friction have been evaluated. The effects of the polyion charge, its size, and the bulk viscosity of the media on the polyion-counterion interactions have also been discussed.

Chapter 3 reports a study on the interactions between a polyion and its counterions in aqueous solutions of another anionic polyelectrolyte sodium polystyrenesulfonate in the presence of NaCl using electrical conductivity as the probe. The specific conductance *vs*. polyelectrolyte concentration data have been analyzed with an equation put forwarded recently[59] considering the scaling description for the conformation of apolyion chain. The influences of (a) the molecular weight and the concentration of the polyelectrolyte, (b) the added electrolyte concentration, and

(c) the temperature on polyion–counterion interactions were investigated. The extent of counterion-condensation was found to be greatly affected by the concentration and molecular weight of the polyelectrolyte, the concentration of the added electrolyte, and the temperature. The polyion equivalent conductivity in conjunction with the derived coefficient of friction between the monomer units and the solvent provided important information concerning the relative importance of the size and charge of the polyions. The overall results have been elucidated taking the relative permittivity of the medium, the hydration behaviour of the counterions, and the coiling behaviour of the polyion chains into account. The present study provides new insight as to how the polyelectrolyte molecular weight influences the counterion-condensation behaviour in a polyelectrolyte solution in the presence of an added electrolyte.

Chapter 4 pertains to a study exploring the influence of sodium carboxymethylcelulose on the aggregation phenomena of a surface active ionic liquid 1-hexadecyl-3-methylimidazolium chloride in aqueous solutions using electrical conductivity and surface tension measurements. The counterion-condensation behaviour of sodium carboxymethylcelulose (aq) and the premicellar ion-association behaviour of 1-hexadecyl-3-methylimidazolium chloride (aq) were also investigated. Two characteristic concentrations, namely the critical aggregation concentration and polymer saturation concentration, before free 1-hexadecyl-3-methylimidazolium chloride micelles appear in sodium carboxymethylcelulose + 1-hexadecyl-3-methylimidazolium chloride solutions, have been identified. Effects of temperature, sodium carboxymethylcellulose concentration, and the charge density parameter of sodium carboxymethylcelulose on the self-aggregation of the 1-hexadecyl-3-methylimidazolium chloride have been discussed to elucidate sodium carboxymethylcelulose + 1-hexadecyl-3-methylimidazolium chloride interactions. The thermodynamic parameters for micellization of 1-hexadecyl-3-methylimidazolium chloride were estimated both in absence and in the presence of sodium carboxymethylcelulose. The observed enthalpy–entropy compensation effect in 1-hexadecyl-3-methylimidazolium chloride and sodium carboxymethylcelulose + 1-hexadecyl-3-methylimidazolium chloride systems provided important insight as to how micellization processes are governed by the bulk structural property of the solution with respect to that of the water.

Chapter 5 gives an account of a study on the aggregation behaviour of the surface active ionic liquids 1-decyl-3-methylimidazolium chloride in aqueous solutions in absence and in presence of sodium carboxymethylcelulose using electrical conductivity, surface tension, vapor pressure, and fluorescence measurements. Ion-association behaviour of 1-decyl-3-methylimidazolium chloride (aq) in the premicellar regime has also been investigated. Two characteristic concentrations, namely the critical aggregation concentration and polymer saturation concentration, before free 1-decyl-3-methylimidazolium chloride micelles appear in sodium carboxymethylcelulose + 1-decyl-3-methylimidazolium chloride solutions were identified. Effects of temperature, sodium carboxymethylcelulose concentration, and the bulk solution structural property on the self-aggregation of 1-decyl-3-methylimidazolium chloride have been discussed to elucidate sodium carboxymethylcelulose + 1-decyl-3-methylimidazolium chloride interactions. Thermodynamics of the micellization processes provided important insight regarding the (a) release of water molecules from the hydration layer around the hydrophilic domain, and from the water cage around the hydrophobic moiety of the surface active ionic liquids, and (b) transfer of the hydrocarbon chains into the micelle and restoration of the H-bonding structure of the water around the micelle.

In **Chapter 6,** the influence of sodium polystyrenesulfonate on the aggregation phenomena of 1-decyl-3-methylimidazolium chloride and 1-hexadecyl-3-methylimidazolium chloride was studied in aqueous solutions using electrical conductivity, surface tension, fluorescence and vapour pressure osmometric measurements. Several characteristic concentrations signifying different kinds of interactions, namely the critical aggregation concentration and polymer saturation concentration, prior to the formation of free surface active ionic liquid micelles in sodium polystyrenesulfonate + surface active ionic liquid solutions have been identified. Effects of alkyl chain length, temperature, sodium polystyrenesulfonate concentration, and the charge density parameter of sodium polystyrenesulfonate on the self-aggregation of the surface active ionic liquids have been discussed to elucidate sodium polystyrenesulfonate + surface active ionic liquid interactions. The thermodynamic parameters for micellization of the surface active ionic liquids were estimated in the presence of sodium polystyrenesulfonate.

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Publications

List of Publications derived from this Study:

1. **D. Ray**, R. De, and B. Das, Thermodynamic, transport and frictional properties in semidilute aqueous sodium carboxymethylcellulose solution, *J. Chem. Thermodyn.*, **101**, 227 (2016).
2. R. De, **D. Ray**, and B. Das, Influence of temperature, added electrolyte, and polymer molecular weight on the counterion-condensation phenomenon in aqueous solution of sodium polystyrenesulfonate: a scaling theory approach, *RSC Adv*ances, **5**, 54890 (2015).
3. **D. Ray**, S. Das, R. De, and B. Das, Sodium carboxymethylcellulose-induced aggregation of 1-decyl-3-methylimidazolium chloride in aqueous solutions, *Carbohydr. Polym.*, **125**, 255 (2015).
4. B. Das, **D. Ray**, and R. De, Influence of sodium carboxymethylcellulose on the aggregation behaviour of aqueous 1-hexadecyl-3-methylimidazolium chloride solutions, *Carbohydr. Polym.*, **113**, 208 (2014).
5. **D. Ray**, and B. Das, Influence of sodium polystyrenesulfonate on the aggregation behaviour of aqueous surface active ionic liquid solutions, *communicated*.

List of other Publications:

6. S. Mondal, **D. Ray,** and B. Das, Thermodynamics of aggregation of imidazolium-based surface activesurface active ionic liquids in aqueous poly(ethylene oxide) media, *J. Chem. Thermodyn.*, **116**, 61 (2018).
7. P. Sar, A. Ghosh, S. Malik, **D. Ray**, B. Das and B. Saha, Selective heteroaromatic nitrogen base promoted chromium(VI) oxidation of isomeric pentanols in aqueous micellar media at room temperature *J. Ind. Eng. Chem.*, **42**, 53 (2016).